

The Ninth U.S.-Japan Seminar on

***Dielectric and Piezoelectric Ceramics***

**PROGRAM AND EVALUATIONS**

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## **U.S. Chairman's Report**

### **Abstract**

The Ninth U.S.-Japan Seminar on Dielectric and Piezoelectric Ceramics was held on Okinawa, Japan, through November 2-5, 1999. The local organization was from the Science University of Tokyo and the Tokyo Institute of Technology. The total number of papers was 106: 69 Japanese and 37 U.S.; 40% of the papers were from industry.

The principal technical topics were: (1) Piezoelectric Ceramics (bulk), (2) Multilayer Capacitors, and (3) Thin Film Dielectrics. The emphasis on multilayer capacitors was on base metal electrodes (BME) with ultra thin layers ( $< 3$  microns) and the search for non-PbO based piezoelectrics a major thrust area in Japan. Novel pyrochlore and quantum ferroelectric materials and improved understanding of polarization fatigue in thin films were highlighted.

### **Participants**

The number of participants of the Ninth U.S.-Japan Seminar were the highest of all the previous meetings, a point of contention, in that "bigger is not better", as commented on by several of the evaluators. Of particular significance, however, was the ~40% attendance rate of U.S. industrial participants, up significantly in contrast to previous meetings, but no major bulk piezoelectric manufacturer was represented.

Table I summarizes the participants as broken down into U.S. vs. Japan and industry vs. university, the latter including government laboratories. Table II summarizes the general topics, with Table III providing a list of industrial participants.

**Table I. Participants of the Ninth U.S.-Japan Seminar on  
Dielectric and Piezoelectric Ceramics**

Total Number of Papers:	106
Japan:	69
U.S.	37
Industry:	
Japan:	26 (~40%)
U.S.:	14 (~40%)
University:	
Japan:	41
U.S.:	26

**Table II. Participants of the U.S.-Japan Seminar by Topic Area**

	<u>Japan</u>		<u>U.S.</u>	
	Industry	University	Industry	University
Piezoelectrics (Bulk)	9	15	3	12
Multilayer Capacitors	9	3	7	4
Thin Films	7	20	2	8
Microwave Dielectrics	2	3	1	1

Misc.: Polymer Packaging (Kyocera)

**Table III. Industrial Participants of the Ninth U.S.-Japan Seminar**

	Japan	U.S.
Piezoelectrics	Ricoh TDK Murata Fuji Electric	Cerone ACX Hewlett Packard
Multilayer Capacitors	Murata TDK Taiyo Yuden Toshiba/Nippon-Chemicon Fuji Titanium	Kemet Degussa TAM Ceramics Ferro Cabot Corp. MRA Labs
Thin Films	Rohm Co. Mitsubishi Materials Fujitsu Seiko Oki Electric Japan Steel Works Ohka Kogyo Co. Sharp Murata	IBM Motorola Radiant Technology
Microwave Dielectrics	Murata Daiken Chemical NGK Spark Plug	Motorola Ferro
Misc.	Kyocera	



# **Program Evaluations**

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# REPORT ON THE 9TH U.S.-JAPAN SEMINAR ON DIELECTRIC & PIEZOELECTRIC CERAMICS

Okinawa, Japan, November 2-5, 1999

by D. M. Smyth, Lehigh University

## A. Introduction

These seminars, which started in 1982, continue to be an excellent opportunity to communicate with our Japanese friends and colleagues, and to learn about new developments and efforts in the field of ceramic dielectrics. They even stimulate further interactions among our own U.S. colleagues. My main interest continues to be focused on multilayer ceramic capacitors (MLCs), especially those with base metal electrodes (BME) such as nickel or copper. This is one of the major technological applications of defect chemistry. In fact, my first exposure to the BME technology occurred during the first of these U.S.-Japan seminars held in Tokyo in 1982. At that meeting Drs. Sakabe and Wakino of Murata disclosed their new reduction resistant dielectric formulation, consisting of an A-site rich, Ca-doped  $\text{BaTiO}_3$ . At that time, the basis for the resistance to chemical reduction of this composition was not clearly understood. From earlier studies of the defect chemistry of  $\text{BaTiO}_3$ , it was clear that the transition from the insulating state to the conducting state is pushed down to more reducing atmospheres by the addition of acceptor dopants. On the assumption that the desired properties require a high degree of acceptor-doping, I proposed that with an excess of what would normally be A-site cations some of the  $\text{Ca}^{++}$  was forced to occupy B-sites, where it functions as a doubly-charged acceptor,  $\text{Ca}_{\text{B}}^{''}$ . We subsequently proved this to be the case, and that the solubility of  $\text{Ca}^{++}$  on the B-sites is about 1.5%, surprisingly high (1,2).  $\text{Ca}^{++}$  will preferentially occupy the A-sites if there is room enough, where it is an isovalent substitution that has almost no effect on any property. But if there is an excess of large cations, some  $\text{Ca}^{++}$  can be forced onto the B-sites, where it is a powerful acceptor and causes a rapid drop in the Curie temperature. In fact,  $\text{Ca}^{++}$  remains the only example of a doubly-charged "amphoteric" dopant, i.e. one that can occupy either of the cation sites in the perovskite structure.

For several years there remained two mysteries about the properties of the Ca-doped formulations. One was how these materials did reasonably well on life tests when the acceptor-doping resulted in a correspondingly large concentration of oxygen vacancies, generally thought to be highly detrimental to life test stability. In fact, the simple doped compositions that we made at Lehigh had very poor life test behavior. I assume that the properties of the commercial formulations are due to the incorporation of additional

dopants that somehow improve the long term stability of the capacitors. This is still not clearly understood.

The other mystery was that the capacitors were routinely fired in such highly reducing atmospheres that I would have expected even these dielectrics to be reduced to the semiconducting state. This was based on the assumption that the shift from insulating to conducting behavior occurs at the transition from predominantly p-type behavior to predominantly n-type behavior, i.e. at the minimum in the equilibrium conductivity measured at high temperatures as a function of oxygen activity. That model assumed that there is no significant trapping of either electrons or holes. However, the lack of trapping of holes is inconsistent with the insulating behavior of p-type  $\text{BaTiO}_3$ . Measurements in our laboratory of the enthalpy of the oxidation reaction by chemical means, rather than by a conductivity measurement, confirmed that hole trapping is extensive, even at the high temperature equilibration conditions (3,4). Thus the transition from insulating to conducting behavior occurs at the shift from predominantly hole species, mostly trapped, to predominantly electron species, all free. This occurs at much lower oxygen activities than that at the conductivity minima, and explains the ability to fire BME capacitors under extremely strong reducing conditions. Moreover, the oxygen activity at the insulator-conductor transition is reduced by the fourth power of the net acceptor excess, rather than as the square dependence of the conductivity minima.

## B. The Present Status and Future of MLCs

Dr. Sasaki of Murata gave an excellent report on the present status and future directions of the MLC industry (5). The growth rate in units produced in recent years has been a phenomenal 25% per year, with worldwide production increasing from 90 billion units in 1992 to 360 billion units in 1998. The bad news is that the price has been declining by 10% annually. The ability to make higher capacitance values has resulted in increasing encroachment into the traditional market for tantalums and aluminum electrolytics. While MLC production increased by 22.4% in the first quarter of 1999, production of aluminum electrolytics declined by 6.4% over the same period. As an example of the larger MLCs, a 100  $\mu\text{f}$  capacitor was described with a volume of less than 0.1  $\text{cm}^3$ , and having 525 dielectric layers, each 3.3  $\mu\text{m}$  in thickness. Dielectric layers of the order of 1  $\mu\text{m}$  are being developed in the laboratory, and such techniques as MOCVD are being explored for even thinner layers. The proportion of MLCs with BMEs has steadily increased; Dr. Sasaki estimated that 80% by weight of the electrode materials currently used in Japan are based on Ni paste. This progression has been driven by several factors: (1) an increase in the price of Pd by a factor of 2.5 in recent years, and (2) the ability to make BME MLCs that meet the more stringent X7R specifications. The latter has been achieved largely by a switch from the traditional use of  $\text{Ca}^{++}$

as the acceptor dopant to rare earth cations, which will be discussed in the next section. L. A. Mann of Kemet described an alternative approach of using high Ag-low Pd electrodes with low-firing dielectric compositions (6). Dr. Mann estimated that at present material costs the break-even point between low-fired and BME capacitors lies at about 0.1  $\mu\text{f}$ , with BMEs being cheaper for higher values. If the price of Pd should return to its more traditional, lower values, the break-even point could shift to about 1  $\mu\text{f}$ .

### C. The Use of Rare Earth Dopants for Capacitors with BMEs

In the last few years, there has been a shift in the doping of BME dielectric formulations from the traditional Ca-doping to the use of certain rare earths. Use of the latter dopants makes it possible to produce BME MLCs that satisfy the X7R and Y5V performance specifications, thus opening up a much wider market. This is of particular interest to me because we worked out the site occupation preferences and resulting defect chemistry of rare earth doped  $\text{BaTiO}_3$  some years ago. This was presented at the Orlando Meeting of the Electronics Division of the American Ceramic Society in 1985, and published in *Advances in Ceramics* in 1987 (7). In this study, the site preference as a function of ionic radius was determined directly by electrical measurement of the acceptor-donor behavior. The shape of the plot of the equilibrium conductivity data as a function of oxygen activity gives direct information on the balance of acceptor-donor behavior.

The rare earths represent a series of trivalent cations that gradually decrease in ionic radii with increasing atomic number as a result of the lanthanide contraction. If the rare earth substitutes for  $\text{Ba}^{2+}$  on the A-sites, it acts as a singly-charged donor, but if it substitutes for  $\text{Ti}^{4+}$  on the B-sites, it acts as a singly-charged acceptor. The experimental data can be superimposed on calculated plots for various site occupation ratios using published mobility data. We found that there is a gradual shift from A-site occupation for the larger ions, e.g.  $\text{Nd}^{3+}$  and  $\text{Sm}^{3+}$ , to B-site occupation for the smaller ions, e.g.  $\text{Yb}^{3+}$ . Cations of intermediate size, e.g.  $\text{Er}^{3+}$ , divided themselves more evenly between the two sites, a behavior that has recently been called "amphoteric". This gradual shift of site preference with ionic radius is at some variance with theoretical calculations by Lewis and Catlow that predicted that self-compensation would favor an equal division between the two sites to a much greater extent than observed (8).

It was also observed that the site occupation could be influenced by the Ba/Ti ratio, especially for the cations of intermediate size. Thus samples doped with  $\text{Er}^{3+}$  appear to be slightly donor doped in the presence of excess Ti, but almost purely acceptor doped in the presence of excess Ba. Clearly the presence of an excess for one site tended to drive the dopant to the other site.

A subsequent paper focused on the behavior of  $\text{Er}^{+3}$  as a dopant, and describes the analysis of the equilibrium conductivity data in greater detail (9). In this work it was found that  $\text{Er}^{+3}$  affected the Curie temperature and room temperature tetragonality only when it was forced onto the B-sites. It was shown quantitatively that  $\text{Er}^{+3}$  occupies only the B-sites in the presence of excess Ba. The solubility of  $\text{Er}^{+3}$  also appeared to be dependent on the Ba/Ti ratio. In an earlier study (10), the series  $\text{Al}^{+3}$ ,  $\text{Sc}^{+3}$ ,  $\text{Y}^{+3}$ , and  $\text{La}^{+3}$ , i.e. the Group III trivalent cations, were similarly studied and the results are mentioned in (7).  $\text{Al}^{+3}$  and  $\text{Sc}^{+3}$  were found to be B-site acceptors,  $\text{La}^{+3}$  an A-site donor, while  $\text{Y}^{+3}$  behaved very much like the amphoteric  $\text{Er}^{+3}$ , which has an almost identical ionic radius.

The preceding discussion is a prelude to mention of the very nice review of rare earth doping given at the meeting by Randall in a plenary talk (11). This group studied the rare earth doped samples by XRD to obtain the unit cell volumes. It is gratifying that the behavior was just what would be predicted from our determination of site occupations, i.e. the larger cations go on the A-sites, the smaller cations go on the B-sites, and the cations of intermediate size go on both sites to varying degrees, and their choice can be affected by the Ba/Ti ratio. Similar conclusions were made by a group from Taiyo Yuden, based on both unit cell size and microstructural observations (12). There were some modest offsets between the findings of the three groups that can be attributed to different processing procedures.

Randall tried to quantify the site-selection process by using a site exchange reaction



This implies that occupation of the B-sites creates barium vacancies, whereas it actually creates oxygen vacancies. It has never been shown that barium vacancies play any significant role in the defect chemistry of  $\text{BaTiO}_3$ . Thus we have shown that in donor-doped compositions, even when formulated to contain the proper amount of compensating barium vacancies, the system rejects this opportunity and splits out sufficient Ti-rich second phase to leave compensating titanium vacancies (13). The reaction shown above, and the resulting mass-action treatment, thus seems to be incomplete. This group also found some effect on site occupation by variations in the oxygen activity, attributed to its effect on the oxygen vacancy concentration. For excess B-site occupation with acceptor-doped behavior, the effect should be small because the oxygen vacancy concentration is primarily fixed by the net acceptor content except under very strong reducing conditions. Even then the vacancy concentration varies only as the sixth root of the oxygen activity.

#### D. Why Rare Earth Dopants?

Finally, it is not clear why the rare earth dopants are advantageous. If their role is to immobilize oxygen vacancies, one would expect  $\text{Ca}^{++}$  to be even better. It is a doubly-charged center instead of singly-charged as are the rare earths, and it is larger than the amphoteric rare earths that are used in MLCs. Thus the rare earths have no advantage for either electrostatic or stress-related mechanisms of vacancy trapping. Secondly, why are the amphoteric rare earths most effective? As a first approximation this just introduces a mix of acceptor and donor centers, so why not just put in a mixture of a pure acceptor and a pure donor, e.g. a combination of  $\text{Al}^{+3}$  and  $\text{La}^{+3}$ ? Moreover, putting some of the dopant on the A-sites reduces the net acceptor content and hence reduces the movement of the insulating-semiconducting transition to lower oxygen activities, which was the purpose of the doping in the first place. The rare earths on A-sites are positive centers that are smaller than  $\text{Ba}^{+2}$  and would then repel oxygen vacancies for both electrostatic and elastic reasons. It has been suggested that the electronic structure of the rare earth ions is important for some reason presumably related to the partially filled 4f shells. Yet  $\text{Y}^{+3}$  also appears to be an effective dopant, and is amphoteric, but has the rare gas electronic structure of krypton and has no f electrons. Thus the peculiar effectiveness of the amphoteric trivalent dopants (certain rare earths and yttrium) in allowing the production of high quality MLCs with BMEs is not at all clear.

#### E. Summary

The development of dielectric compositions that can be fired under reducing conditions so that the expensive Ag-Pd electrodes can be replaced by Ni has been a prominent and interesting application of defect chemistry. This has kept the materials costs down so that these capacitors can effectively compete with alternative types. The improved properties that have been achieved by the replacement of the traditional  $\text{Ca}^{++}$  dopant with the amphoteric rare earths have opened up an even wider market. While the basic behavior and site occupation choices for the rare earth dopants have been known for over 10 years, the reasons for the improved stability of capacitors with these newer dopants remains unclear.

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**9th Bi-annual US-Japan Seminar on Dielectric and  
Piezoelectric Ceramics**

**Nov 3-5, 1999**

Okinawa, Japan

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In addition to the main conference in Okinawa, I visited Toshiba Corporation's Central Research facility on October 29.

**Visit to Toshiba Corporation's Central Research Facility**

The visit to Toshiba was organized through Yoshiki Ishizuka, who worked as a visiting scientist at North Carolina State University primarily in 1998. The official host for the Toshiba visit was Dr. Noburu Fukushima. The work of Dr. Fukushima most closely mirrors that of our group at NC State thus he was the most appropriate person.



I arrived on Thursday afternoon at Narita airport and stayed the evening at the Nikko hotel in Kawasaki. Arrangements were made for me by Mr. Ishizuka. I arrived at Toshiba at 10 am, the following list details the days agenda.

time	function	Toshiba participant
10:00	greetings and general overview	Dr. A. Toriumi
		Dr. Kurobe
10:15	Presentations by Toshiba	
	Ta-oxide gate dielectrics	Dr. Y. Tsunashima
	Epitaxial BST capacitors	Dr. K. Abe
12:00	Lunch	
13:00	Presentation by Visitor	Dr. J-P. Maria
	Alternative gate dielectrics	
15:00	Presentations by Toshiba	
	New gate dielectrics	Dr. A. Nishiyama
	MFIS FETs	Dr. S. Takagi
	PZT FERAMs	Dr. K. Yamakawa
17:00	Closing discussions	all participants

My presentation at Toshiba focused on current research aimed at identification and development of alternative gate dielectrics for silicon transistors. In addition, a general overview of research in our group at NC State was presented. All materials were well received. Toshiba's research and presentations on BST capacitors was particularly impressive. They appear to be close to implementation of epitaxial capacitors in high permittivity embedded DRAM devices. In the field of gate oxides, however, the Japanese community appears to be several years behind the United States. Many Japanese researchers are still focusing on  $Ta_2O_5$ , a material which main stream researchers in the US have largely abandoned for multiple reasons.

After the Toshiba presentations and discussions, Mr. Ishizuka acted as a host for the entire evening and the next afternoon. Mr. Ishizuka paid for excellent dinners both Friday and Saturday evenings and provided me with an excellent tour of the Tokyo area.

#### **November 3<sup>rd</sup> - 5<sup>th</sup> US-Japan Seminar**

- Traveled from Tokyo to Kyoto and met Professor David Cann of the Iowa State University. Professor Cann and I spent a day touring Kyoto then traveled together from the Kansai airport to Okinawa.

- Registered for the seminar on the evening of November 2 and attended the reception. The meeting chairs, Dr. ShROUT and Dr. Takenaka, did an excellent job making preparations. In general, all social activities associated with the conference were organized similarly.

- Attended the seminar for the next three days, the following section discusses the noteworthy presentations made in the general meeting.

### **Session I - Basic Science**

- "MLC technologies of today and future" Yukio Sakabe, Murata Manufacturing Inc. Dr. Sakabe gave an insightful presentation detailing the recent developments of Murata's capacitor research. The specific topics of note were rare earth doping of MLCs with base metal electrodes and thin film MLCs made by MOCVD. The thin film MLC work was rather impressive especially in the context of the number of layers (15) and the remarkable smoothness of even the top levels.
- "Ferroelectricity in  $\text{SrTi}(\text{O}^{16}\text{O}^{18})$ " by M. Itoh. This talk was of great interest as it demonstrated the ability to induce a ferroelectric phase transition in the normally incipient ferroelectric  $\text{SrTiO}_3$  with the simple substitution of an oxygen isotope. Though no mechanistic details were presented, the results were compelling.

### **Session II - Piezoelectric materials and devices**

- "High piezoelectric performance in barium titanate single crystals with engineered domain configurations", S. Wada. Professor Wada presented a delightful poster which discussed the ability to engineer the domain configurations of ferroelectric materials when specific, and

unexpected, measurement orientations were used. Of particular interest was the ability to find orientations where the dielectric loss and piezoelectric hysteresis values were exceptionally small. Wada also suggested that this behavior could be true for all ferroelectric perovskites of appropriate symmetry.

- "Fatigue anisotropy for rhombohedral PZN-PT single crystals", K. Takemura This work from Penn State represents the latest breakthrough in the understanding of polarization fatigue for ferroelectric materials. Takemura et al. found a specific orientation dependence to fatigue suggesting an additional link to preferential domain switching.

### **Session III - Thin film dielectrics**

- "The electrical properties of thin barium strontium titanate films and their impact on the performance of capacitors for DRAM memories", T. Shaw. Dr. Shaw presented a discussion detailing the recent developments for BST embedded DRAM memories. In depth discussions of strain and composition effects were given which explained the possibilities and limitations for DRAM based on paraelectric barium strontium titanate.
- "Synthesis of new pyrochlore compounds for transparent conductor applications", D. P. Cann. Professor Cann prepared a poster discussing his recent efforts at producing new transparent conducting oxides. Of particular interest to Dr. Cann is the ability to make a family of

transparent conductors whose band gap can be compositionally engineered. This work is of technological importance for optical devices operating at increasingly short wavelengths.

#### **Session IV - Multilayer ceramic capacitors**

- "Dielectric property of  $\text{BaTiO}_3\text{-BaZrO}_3$  solid solution under high electric field". T. Tsurumi.

Dr. Tsurumi's work investigated the high field behavior of the BTO-BZO system. Of particular importance was the realization that the relaxor-like characteristics of such systems must be considered when interpreting the high voltage characteristics. The ability to shift the ferroelectric transition with field gives such materials a false voltage dependence of the dielectric constant. This behavior must be well understood for reliable implementation into electronic components, especially those targeted for high frequency or high power applications.

#### **Summary**

The 9<sup>th</sup> US-Japan seminar was of high technical quality and well managed. Organizers from both sides did well to select an appropriate industrial and university cross section. The conference participants represented a good mix of research topics currently of great scientific interest, as well as those of industrial and economic importance. This was the second US-Japan seminar that I attended. Once again, I have very positive reviews and intend to participate in future years.

## **9th U.S. Japan Seminar on Dielectric and Piezoelectric Ceramics**

November 2-4 1999

Okinawa, Japan

David P. Cann

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### **US-Japan Seminar on Dielectric and Piezoelectric Ceramics**

I arrived in Okinawa on Tuesday afternoon on a flight from the new Osaka Kansai airport. After registering for the meeting in the afternoon, I attended the reception which was well attended. The following mornings session on basic science was highlighted by the plenary lectures by Clive Randall and Yukio Sakabe. The rest of the morning session consisted of talks concerning a number of topics including ferroelectricity, fatigue resistance, and a number of talks on PZT. The most interesting talk came from M. Itoh and R. Wang from the Tokyo Institute of Technology on the effects of  $^{18}\text{O}$  stoichiometry in  $\text{SrTiO}_3$ . In their dielectric measurements they showed a strong relationship between the maxima in permittivity and  $^{18}\text{O}$  content at cryogenic temperatures. The afternoon session on piezoelectrics had a total of 26 presentations on various facets of piezoelectric materials, devices, and applications.

The Thursday morning session on thin films was highlighted by a presentation by Thomas Shaw of the IBM Microelectronics Division on thin film BST capacitors for DRAM applications. Of the 22 other papers presented in this session a significant fraction (11) was devoted to thin films of PZT. A total of four papers focused on  $\text{SrBi}_2\text{Ta}_2\text{O}_9$ , 2 papers on PMN-PT, and the rest included pyrochlores,  $\text{PbTiO}_3$ ,  $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ , and  $\text{Ba}(\text{Sn},\text{Mg},\text{Ta})\text{O}_3$ . In this session I presented the recent results of our group on new compounds with the pyrochlore structure which are aimed at transparent conducting oxide applications. One of the more interesting talks was from H. Tamura *et al* of Murata Manufacturing, on microwave measurements of HTSC electrodes on  $\text{Ba}(\text{Mg},\text{Sn},\text{Ta})\text{O}_3$  dielectric resonators. Large Q values for the dielectric resonators made with HTSC electrodes were 7 times higher than Ag were recorded, but microstructural problems limit the high frequency and high power applications.

Thursday afternoons presentations were focused on multilayer ceramic capacitors. The plenary talk by Larry Mann of Kemet compared different multilayer capacitor technologies. Using dielectric breakdown, mechanical strength, and life tests as a comparison, he found that low-fire Ag-based and high-fire Ni-based capacitors showed similar performance characteristics. Other talks in this session investigated the role of rare-earth element doping in  $\text{BaTiO}_3$  and the use of nano-sized powders in thick film processing.

The conference ended with a session on advanced packaging and processing. This session included discussions on chemical preparation routes for  $\text{BaTiO}_3$ ,  $(\text{Pb,Ba})\text{Nb}_2\text{O}_6$ , and PZT among others.

I left immediately after the morning session flying back to Osaka, and then taking the Shinkansen to the Tokyo-area. The weekend following the conference was spent visiting with former Japanese colleagues including Dr. Koji Yamakawa of Toshiba, Mr. Nakano Atsushi and Mr. Hitomi Atsushi of TDK Corporation, and Dr. Satoshi Wada of the Tokyo Institute of Technology.

#### **TDK Materials Research Center, Narita:**

On the Sunday following the US-Japan meeting, I arrived in Narita with Mr. Hitomi Atsushi and Mr. Nakano Atsuyuki of TDK. After spending the night in Narita, the following morning I visited TDK's Materials Research Center facility. I gave an informal presentation to the researchers from the ceramic R&D group including Takeshi Takahashi and Dr. Christopher Williams (a visiting scientist from Cranfield). I presented recent work on the following:

- (i) pyrochlore oxides for transparent conducting coating applications
- (ii) microelectrodes fabricated via focused ion beam (FIB) milling



(iii) isotropic negative thermal expansion zirconium tungstate

I received valuable comments and suggestions from the audience on all three topics. One idea that was borne out of our discussion was to look at the possibility of ionic conduction in zirconium tungstate. Due to the open structure it is highly possible that there are channels for conduction. There has been one report of possible oxygen conduction in  $\text{ZrW}_2\text{O}_8$ . The interesting thing about ion conduction in  $\text{ZrW}_2\text{O}_8$  is that because of the negative thermal expansion the material may well have a negative thermal coefficient of ionic conductivity unlike most other known ionic conductors.

After my presentation and the subsequent discussion I received a brief tour of their facilities. Afterwards, TDK hosted a dinner at a local restaurant with Clive Randall and Tom Shrout of Penn State who were visiting a different facility at the same time.

### **Mitsubishi Materials Central Research Institute, Omiya**

On Tuesday morning the 9th of November I traveled to Omiya to visit Mitsubishi Materials Central Research Institute. I was invited by Dr. Kuromitsu Yoshirou who was a visiting scientist at the Materials Research Lab at Penn State while I was in graduate school. Immediately following my arrival, Dr. Kuromitsu gave me a tour of their

facilities. They have a wide range of expertise from powders, to magnetic materials, to his current program on flat panel display technologies.

After having lunch with one of his coworkers Mr. Hideaki Sakurai I gave a presentation on the same topic that I gave at TDK. The group was especially interested in the transparent conducting oxide work due to their strong focus on display technologies. After the meeting I spoke with members of the flat panel display group on a variety of topics including wide band gap materials, low firing temperature conductors using nanopowders, and suppression of secondary electron emission. Overall, I was impressed with the quality of their processing technologies. With their current schemes, they are able to get 100% yield on their 42 inch displays which use 0.2 mm cells. What is most impressive is the number of layers in their device structure. Having such a high yield over such a large surface is unlike anything else I know of in electronic ceramics. Following my day at Mitsubishi Materials, I went to dinner with Dr. Kuromitsu and his family.

The following day I returned to the US after a total of 9 full days in Japan.

### **Summary**

In summary, I found the US-Japan meeting to be stimulating, very well organized and was set in a beautiful setting in Okinawa. The conference organizer from Japan Dr.

Tadashi Takenaka in his opening remarks presented data illustrating of the increase in size of the meeting over the years. I hope the meeting does not grow too large as I think one of the most unique aspects of the meeting is the ease with which researchers can meet and discuss their work in an informal relaxed atmosphere. This is the second US-Japan meeting that I have attended and both have been amongst the best scientific conferences I have attended.

The visits to TDK and Mitsubishi were also extremely insightful and well worth the trip alone. In conclusion, I would like to thank the Office fo Naval Research for sponsoring my trip.

## Travel-Report for the 9<sup>th</sup> US-Japan Seminar on Dielectric & Piezoelectric Ceramics

Herbert Giesche

NYSCC at Alfred University

### General Comments

The organizer had chosen an appropriate location for that meeting. However, It would have been nice to have some more social interaction. Besides the evening dinners there was not too much possibilities to have a closer contact with some colleges. I have attended two similar meetings/conferences in the past. They had been organized in a slightly different way. Each day a general topic was given and after dinner the group broke out into smaller discussion groups. After about 1 \_ to 2 hours the groups came back together and the results of each sub-group were presented to the entire group. The interesting part of this exercise was the fact that a real discussion occurred instead of just the presentation of research results. It also gave younger students (and faculty) the chance to get a better feeling for the current research trends. Most people had been very free and open to discuss not just what problems they have already solved but actually described where they saw problems for which they did not have a ready answer or where the research would be going in the next couple of years. The careful selection of leading scientists in the respective areas and distributing them among the groups ensured that a good balance was achieved. In my opinion those evening activities had a tremendous effect on the overall success of the meeting. I would also recommend that the maximum size for this kind of meeting should be 100 people or rather 80.

I was extremely impressed by the generous support and other contributions from several of the Japanese companies.

## Comments about the Scientific Program

The recent US-Japan seminar was the first meeting, which I attended in that scientific area. I got only recently more involved in the area of electronic ceramic materials. I have presently two projects that study the preparation of nanosized barium titanate powders by a microemulsion synthesis technique. Thus, pretty much all talks and presentations were new for myself. The meeting certainly helped me to gain a general understanding about the present activities and seeing where there is a need for further improvements in this area. I was especially interested in presentations, which focused on unique properties on the basis of the chemical composition of the materials, since my own background is in chemistry. Moreover, I was very interested in all kinds of powder synthesis techniques or powder processing routes. From that point of view, especially the first session with the plenary lectures by Yukio Sakabe and Clive Randall and also session V on advanced processing and packaging had numerous highly interesting presentations.

From the large list of presentation I would highlight several of them, which were most interesting for myself.

First there had been two presentations by Rayner and Whatmore, showing the application of a piezoelectric device in a flexension motor as well as a traveling wave motor. Those examples could certainly be extremely useful for any kind of miniaturized mechanical devices or any device that would fall in the category of "lab on the chip". A second area was the two plenary lectures by Thomas Shaw and Hidemi Takasu. Both of them focused on the application in memory devices. The connection point to my own work was here the scale down of device features into the nanometer size range. Any properties influenced by the nanometer sized features can right away translated into properties expected in nanosized powders. In session IV the plenary talk by L.A. Mann was very interesting since it provided again for myself a connection to nano sized materials. Mann presented advances in low fire dielectric technology. The following poster session had several interesting contributions. I would like to mention here only the presentation by Gupta et al on chemically prepared barium titanate MLCC and the presentation by N.

Ogata et al on the development of nanosized silver metal platelets and the development of an electrophoretic deposition process to form electrode layers in thin layered dielectrics. The latter presentation was describing a very similar synthesis process, microemulsion synthesis, as what I am presently using for the preparation of the barium titanate particles and it also used electrophoretic deposition for the formation of layer structures. This is very similar to plans in my own research project and I had a very fruitful discussion with the author. I will most probably use this contact to develop a stronger interaction between my own research group and the corresponding group at Penn State (Jim Adair & Clive Randall).


Overall it was in some cases too much information for myself, which can certainly be attributed to my own lack of knowledge, but the meeting helped to give me new ideas and contact addresses for future cooperation. I am very interested to attend the next meeting of that series and hope that at that time my own research has given me a much better basis for a detailed discussions.

### **ONR Trip Report:**

The author attended the 9th US-Japan Seminar on Dielectric & Piezoelectric Ceramics from 2-5 November 1999 to and presented an invited talk.

While in Japan, he also visited on 8 November 1999 Taiyo Yuden Co. Ltd. in Gunma, a major producer of electroceramic components, where he was hosted by Dr. Masayuki Fujimoto. A second visit was made on 12 November 1999 to Panasonic's corporate research laboratories in Osaka, where a presentation was made on his piezoelectric research. Both of the companies visited expressed interest in developing lead-free substitutes for PZT and other lead oxide based dielectrics and piezoelectrics. Interestingly, the driving force for this was not considered to be new regulations in Japan, but in the European market into which both companies sell.

In between these 2 company visits, the author attended the Kyoto Prize Ceremonies in Kyoto, where W. David Kingery was honored as this year's laureate in Advanced Technology.

  
Yet-Ming Chiang  
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**9<sup>TH</sup> US-JAPAN SEMINAR ON  
DIELECTRIC AND PIEZOELECTRIC CERAMICS  
November 2-5, 1999  
Okinawa, Japan**

**PROGRAM**

**TUESDAY, NOVEMBER 2**

**18:00**      *Registration and Welcome Reception*

**WEDNESDAY, NOVEMBER 3**

**8:45-9:00**      Opening Remarks: T. Takenaka (*Science University of Tokyo, Japan*)

***Session I - Basic Science***

Session Chair: Susan Troler-McKinstry, The Pennsylvania State University  
Takashi Yamamoto, National Defense Academy

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I-3	SHG Microscope: Principle and Its Application to Nondestructive Observation of 180°, Domain Structure and Domain Reversal Process in Ferroelectrics, Haruyuki Mohri, Sunao Kurimura and <u>Yoshiaki Uesu</u> , Department of Physics, Waseda University, Japan	23
I-4	(Ba,Sr)TiO <sub>3</sub> Dielectrics: Relationship between Bulk and Thin Film Properties, <u>Angus I. Kingon</u> and Charles B. Parker, Dept. of Materials Science and Engineering, North Carolina State University; Stephen K. Streiffer, Argonne National Laboratories; and Susanne Stemmer, University of Illinois, USA	27



I-5	Quantum Paraelectricity in Epitaxial Titanate Perovskites, <u>R.M.V.Rao</u> , <u>K.Shimada</u> , <u>M.Lippmaa</u> , <u>M.Kawasaki</u> , <u>Y.Inaguma</u> , <u>M.Itoh</u> , <u>H.Munekata</u> and <u>H. Koinuma</u> , Tokyo Institute of Technology, Japan	31
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I-10	Ferroelectricity-Evoking Mass-Inequality Factor for Perovskite Titanates $\text{ATiO}_3$ , <u>Tetsuro Nakamura</u> , Yue Jin Shan, Utsunomiya University; Mitsuru Itoh and Yoshiyuki Inaguma, Tokyo Institute of Technology, Japan	51
I-11	Dielectric Properties and Depoling Characteristics of $\text{PB}(\text{Zr}_{0.05}\text{Ti}_{0.05})\text{O}_3$ Based Ceramics: Near-Critical Grain Size Behavior, <u>B.A. Tuttle</u> , <u>J.A. Voigt</u> , <u>T.W. Scofield</u> , <u>P. Yang</u> , <u>D.H. Zeuch</u> , and <u>M.A. Rodriguez</u> , Sandia National Laboratories, USA	55
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Akira Ando, Murata Manufacturing Co., Ltd.

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- II-2 Crystallographically Engineered Single Crystals for High Performance Piezoelectrics, Seung-Eek Park, Satoshi Wada\*, Paul Rehrig, Shi-Fang Liu, L. Eric Cross, and Thomas R. Shrout, The Pennsylvania State University, USA; \*Tokyo University of Agriculture and Technology, Japan. **87**
- II-3 Non-180° Domain Contribution to the Properties of PZN-PT Single Crystals, Takaaki Tsurumi, Keishiro Okamoto, Naoki Ohashi, Tokyo Institute of Technology; and Yohachi Yamashita, Toshiba corp., Japan **91**
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- II-8 Crystal Growth and Characterization of New Langasite-type Compounds for Piezoelectric Applications, Kiyoshi Shimamura, Tomohiko Kato, Jun Sato and Tsuguo Fukuda, Tohoku University, Japan **111**
- II-9 Crystalline Structure and Piezoelectric Properties of Bi Layer Structured Compound  $\text{SrBi}_2\text{Nb}_2\text{O}_9$ , Akira Ando, Masahiko Kimura and Yukio Sakabe, Murata Manufacturing Company Limited, Japan **115**
- II-10 The Thickness-Extensional and Thickness-Shear Vibration Mode Characteristics of Bismuth Layer-Structure Compounds, Hitoshi Oka, Masakazu Hirose, Takeo Tsukada, Keisuke Itakura and Yasuharu Mjiyauchi, TDK Corporation, Japan **119**
- II-11 Additive Effects on Piezoelectric Properties of  $(\text{Bi}_{1/2}\text{Na}_{1/2})\text{TiO}_3$  Ceramics, Hajime Nagata, Sinichi Morita, Akihiro Itoh and Tadashi Takenaka, Science University of Tokyo, Japan **123**
- II-12 Processing and Piezoelectric Properties of  $\text{Pb}(\text{Ni}_{1/3}\text{Nb}_{2/3})\text{O}_3\text{-PbTiO}_3\text{-PbZrO}_3$  Solid Solutions from PbO-excess Compositions, Keiji Kusumoto and Tadashi Sekiya, National Industrial Research Institute of Nagoya, Japan **127**
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- II-23 The Dynamic Analysis of Kyser-type Ink-Jet Head, Yujiro Kitaide, Fuji Electric corporate R&D, Ltd, Japan 171
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Hiroshi Kishi, Taiyo Yuden Co., Ltd.

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Tadashi Takenaka, Science University of Tokyo

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